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TRANSPARENT, POLYCRYSTALLINE CUBIC ALUMINUM OXIDE

JAMES W. McCAULEY and NORMAND D. CORBIN
CERAMICS RESEARCH DIVISION

September 1980

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ABSTRACT

The stringent mechanical/thermal and broadband electromagnetic wave transmission requirements for sensing mechanisms of the future require optimized and new material concepts. Aluminum oxide-based ceramics and single crystals are strong contenders for these applications, but exhibit significant directional variation (anisotropy) in properties. This barrier to utilization has been eliminated by an AMMRC-invented material - sintered polycrystalline nitrogen-stabilized cubic aluminum oxide (ALON). This material has been fabricated into dense, transparent bodies with *isotropic* properties: Knoop (100) hardness of 1800, elastic modulus of 46×10^6 psi, a dielectric constant and loss tangent, respectively, at 10 MHz of 8.56 and 0.0004, trivial oxidation in air up to 1200°C, an IR cutoff at 5.2 μm, and an average thermal expansion coefficient (α) of 7.00×10^{-6} C^{-1} (25°C-1000°C). These properties suggest greatly improved performance in many other Al_2O_3 applications.

Successful fabrication of ALON was preceded by the determination of the high temperature phase equilibria and crystal chemistry of aluminum oxynitride spinels in the Al_2O_3-AlN system.

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CONTENTS

I. INTRODUCTION

The means used to observe or sense the enemy have progressed from actual eye-to-eye observation to extensive use of radar and sonar, and now include using infrared (IR) signals. At the same time, various forms of armor, from face shields to sophisticated electromagnetic (EM) windows and domes (radomes, IR domes), have been developed to transmit signals and also to protect the sensing mechanisms - either the human eye or intricate electronic devices. Countermeasures such as smoke and radar-jamming systems have concurrently evolved to defeat the various sensing devices. In order to minimize the effectiveness of dedicated (single-mode) or even broadband countermeasure tactics, sensing devices of the future, therefore, must be able to simultaneously function over a large region of the EM spectrum, including visible light, IR, microwave and millimeter wave radars. It is imperative, then, that new materials must be developed to transmit a wide range of the EM spectrum, while at the same time protecting the fragile sensing equipment in wide-ranging types of severe battlefield environments. Consideration of the above criteria results in the conclusion that only a select few materials, if any, can provide all the requirements. Further, the material should be isotropic to minimize any distortion of the transmitted signal. In lieu of isotropic materials, recourse has been made to the use of single crystals of anisotropic materials, oriented to transmit the signals in isotropic or near-isotropic direction. Aluminum oxide is a candidate material for these applications, but has been neglected from serious consideration because of its property anisotropy, relatively high coefficient of thermal expansion, and high cost for the production of single-crystal shapes (1). True optical transparency of polycrystalline Al_2O_3 is impossible, unless all the grains are identically oriented. Further, significant strain can result at grain boundaries due to thermal expansion mismatch of misoriented grains (2). An alternate approach to this inherent problem is the stabilization of a cubic Al_2O_3 structure. A defect cubic spinel, γ-Al_2O_3, (3) can be prepared in powder form, but fully dense ceramics have not been reported due to the ease of conversion to the more stable alpha form at moderate temperatures ($\sim$1000 C) (4,5). However, it has been known for some time (6) that nitrogen additions to Al_2O_3 in the form of AlN can produce spinel-like structures. Since that time various efforts have

1. **BARATA, J., and GORNI, J.** ***Improvements in Mechanical and Electrical Properties of Alumina.*** **Powder Metallurgy International, v. 4, no. 3, 1972, p. 124-128 and no. 4, p. 201-203.**
2. **CAMPBELL, W. J., and GRAIN, C.** ***Thermal Expansion of Alpha Alumina.*** **Bureau of Mines Report of Investigation No. 5757, 1961, 16 pages.**
3. **WEFERS, K., and BELL, G. M.** ***Oxides and Hydroxides of Aluminum.*** **Technical Paper No. 19, Alcoa Research Laboratories, 1972, 51 pages.**
4. **JELLINEK, M. H., and FANKUCHEN, I.** ***X-Ray Diffraction Examination of Gamma Alumina.*** **Ind. and Eng. Chem., v. 37, no. 2, 1945, p. 158-163.**
5. **STEINER, C. J. P., HASSELMAN, D. P. H., and SPRIGGS, R. H.** ***Kinetics of the Gamma-to-Alpha Alumina Phase Transformation.*** **J. Am. Ceram. Soc., v. 54, no. 8, 1971, p. 412-413.**
6. **ADAMS, I., AU COIN, T. R., and WOLFF, G. A.** ***Luminescence in the System Al_2O_3-AlN.*** **J. Electrochem. Soc., v. 109, 1962, p. 1050-1054.**

been made to understand the phase equilibria in this system (7-9). The phase diagrams do not, however, indicate the temperature limits of stability for the γ-Al_2O_3 type oxynitride material, nor has single-phase material been successfully sintered. This report describes the results of a program concerned with refining the temperature-composition stability limits of cubic aluminum oxynitride spinel (ALON - nitrogen-stabilized cubic aluminum oxide) so that fully dense, single-phase ceramics could be sintered.

II. EXPERIMENTAL

Sintering and phase equilibria studies were carried out in an inductively heated graphite furnace using flowing N_2 (1/2 liter per minute at about 1 atm). The starting powders of γ-Al_2O_3* (1.1 μm at 50%) and AlN* (14 μm at 50%) were ball-milled for 24 hours using an ethanol fluid media, isostatically pressed at 25,000 psi, and pre-reacted at 1200 C for 24 hours in gas tight flowing N_2, prior to final sintering studies. Only small amounts of impurities were picked up in the ball-milling procedure. However, more sophisticated sintering studies would require an improved mixing procedure.

Final reaction and sintering runs at elevated temperatures were conducted for one hour so that direct comparison between runs could be made. Weight loss was determined for each specimen and each was characterized by X-ray diffraction and reflected light microscopy. Density measurements and transmitted light microscopy were also carried out on selected products. Our fundamental premise is that we are attempting to deduce the high temperature equilibrium relations and sintering mechanisms from the resultant products. Without the use of sophisticated apparatus, volatility is extremely difficult to suppress, so quantitative measurements of weight loss will yield useful information on vapor phase formation. The reaction samples were contained in a covered BN crucible with a sight hole for pyrometric temperature measurement. Neutron activation analysis of the AlN indicated 1.7 wt% (∿1.5 mole % Al_2O_3) oxygen. The AlN powder also contained about 1 to 2 wt % of unnitrided Al metal powder.

III. RESULTS AND DISCUSSION

Figure 1 illustrates the temperature-composition stability limits for cubic aluminum oxynitride spinel (ALON) in the Al_2O_3-AlN system for 1 atm of flowing N_2 gas. The phase relationships were deduced from careful analyses of both microstructural and X-ray diffraction data. The Al_2O_3-AlN system is a pseudobinary composition

*Cerac/Pure Inc., Butler, Wisconsin

7. LEJUS, A. *Formation at High Temperature of Nonstoichiometric Spinels and of Derived Phases in Several Oxide Systems Based on Alumina and in the System Alumina-Aluminum Nitride.* Rev. Int. Hautes Temp. Refract., v. 1, no. 1, 1964, p. 53-95.
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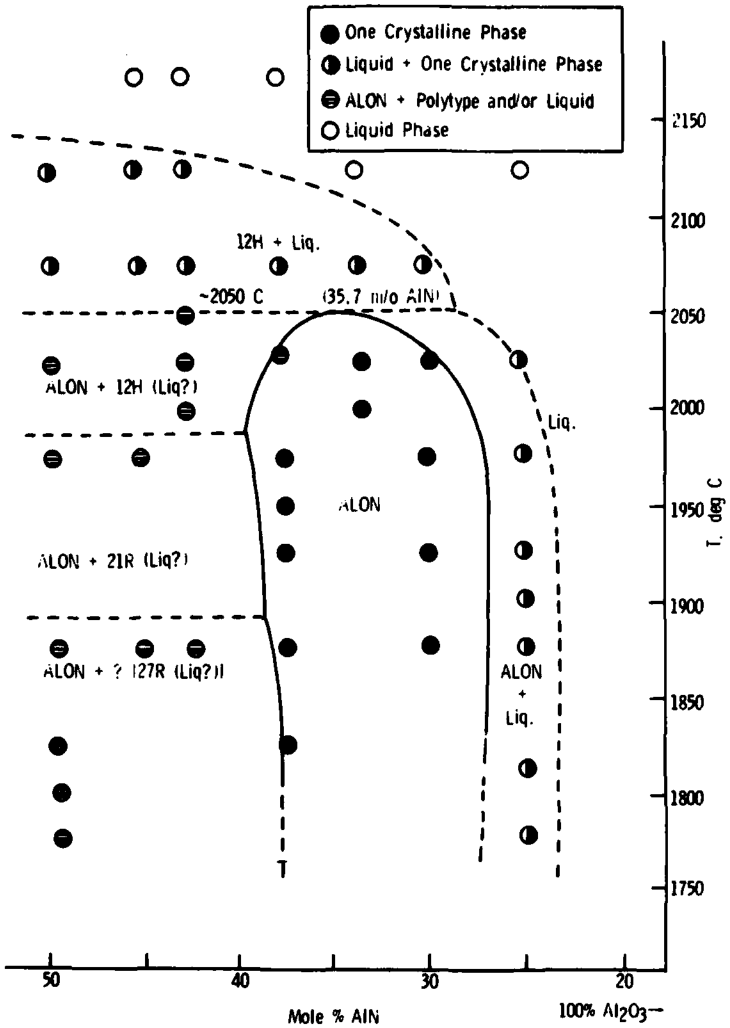


Figure 1. Proposed isobaric (1 atm of flowing N_2) high-temperature phase relationships in the region of ALON stability in the pseudobinary Al_2O_3-AlN composition join.

join in the Al-N-O system. Hence, the phase rule allows for phase fields with up to three coexisting phases. The designations 12H, 21R, and 27R refer to the so-called "AlN" polytypes (10). In this system there seems to be an intimate relationship between liquid formation and the appearance of the various polytypes. Further, the morphology of the polytypes are variable and seem to reflect the difficulty in attainment of equilibrium. It is our conclusion from this work in the vicinity of the ALON stability field and also other parts of this system that some of the polytypes are metastable products of quenched or poorly quenched liquids. We have not yet determined how to differentiate between AlN polytypes which are metastable from those which are not. Hence, in Figure 1 we have "dashed in" all the phase boundaries dealing with polytypes and in some cases have not differentiated between a liquid and the polytypes.

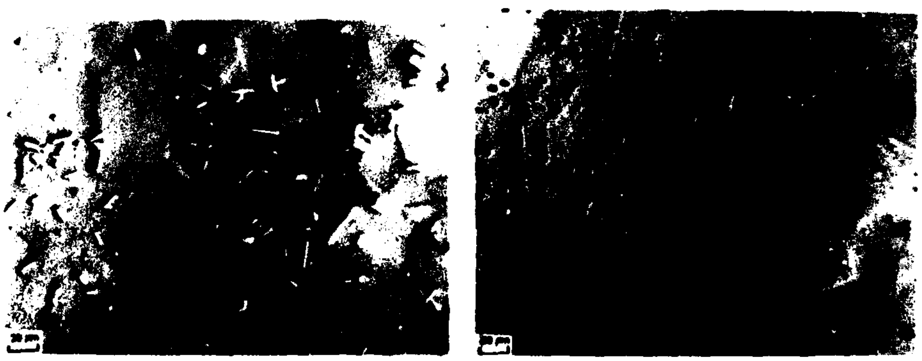
10. JACK, K. H. *Sialons and Related Nitrogen Ceramics.* Review, J. Materials Sci., v. 11, 1976, p. 1135-1158.

There is a relatively wide range of compositional stability, roughly centered at 35.7 mole % AlN, and a maximum in thermal stability at about 2050 C. At this point ALON seems to melt incongruently into one alumina-rich, stable liquid and one nitride-rich, unstable (volatile) liquid. At about 2000 C on the AlN-rich side of the single-phase field, vaporization increases dramatically which kinetically seems to influence reactions in the single-phase region.

As previously indicated by McCauley (11) a constant anion spinel model seems to predict an ALON composition at 35.7 mole % AlN. Using the chemical formula $Al_{(64+x)/3} \square_{(8-x)/3} O_{32-x} N_x$ obtained from this model, the following composition for N = 5 can be calculated:

$$Al_{23}O_{27}N_5 \equiv 5\ AlN \cdot 9\ Al_2O_3.$$

Crystalline solution stability limits were determined by detailed reflected light microscopy and refined lattice parameters. Figure 2 illustrates the phase assemblages on the 37.5 mole % AlN composition line on either side of an apparent phase boundary line between the ALON single-phase field and the liquid plus ALON and 12H polytype (and/or liquid) region. Note the dramatic disappearance of porosity in Figure 2a and the concurrent appearance of liquid. The liquid and 12H polytype appear as the lighter colored intergranular phases in Figure 2a; in Figure 2b the darker circular areas are remnant porosity. This liquid occasionally quenches to a noncrystalline phase, but also crystallizes into various types of AlN polytypes, in this case the 12H polytype. Figure 3 illustrates the microstructure of the ALON plus liquid region on the Al_2O_3-rich side of the single-phase field.



a. 2025 C - in liquid + ALON(SS) field; 10.04 wt% loss

b. 1975 C - in ALON(SS) field; 2.88 wt% loss

Figure 2. Microstructures of phase assemblages in the Al_2O_3-AlN system; 37.5 mole % AlN.
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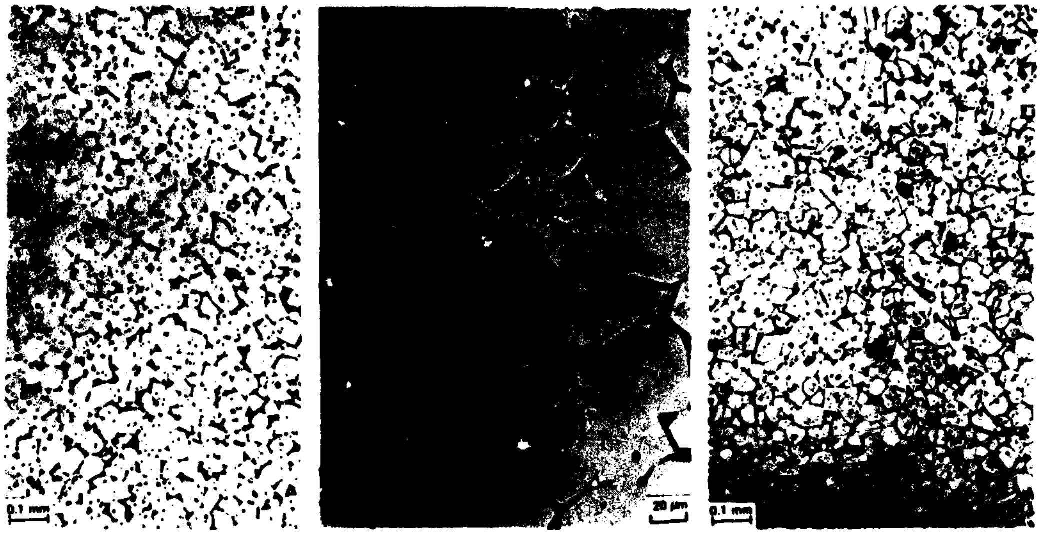
11. McCAULEY, J. W. *A Simple Model for Aluminum Oxynitride Spinels.* J. Am. Ceram. Soc., v. 61, nos. 7-8, 1978, p. 372-373.



a. ALON(SS) + liquid   b. ALON(SS) + liquid   c. Etched

Figure 3. Microstructures of phase assemblages in the Al_2O_3-AlN system; 25 mole % AlN; T = 2025 C.

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In order to confirm the microstructurally determined range of ALON crystalline solution, refined lattice parameters were determined by a least-squares technique on X-ray powder diffracting data. These data are illustrated in Figure 4 along with those obtained by Lejus (7) on material fabricated at 1700 C. There is about a five mole % AlN difference between a least-squares line through the 1975 C data and the Lejus line. The difference can possibly be ascribed to a higher oxygen content of the latter material or the sluggish reaction rates at 1700 C. The equation for this line is indicated on the figure.

ALON ceramics of various nitrogen compositions can be sintered using the experimental conditions previously indicated to 99% of theoretical density. The theoretical densities were calculated using the constant anion model and the refined lattice parameters. Figure 5 shows a typical set of characterization data on a series of runs at 37.5 mole % AlN. By relating these data to the microstructures illustrated in Figure 2 it can be seen that the appearance of liquid results in a dramatic increase in volatility, represented by weight percent loss on the figure; percentage of theoretical density is also indicated in parentheses. A reaction and sintering scheme is also indicated on the figure.

Reaction and Sintering → Sintering (No Liquid) →

Sintering (Liquid + Vapor Formation)

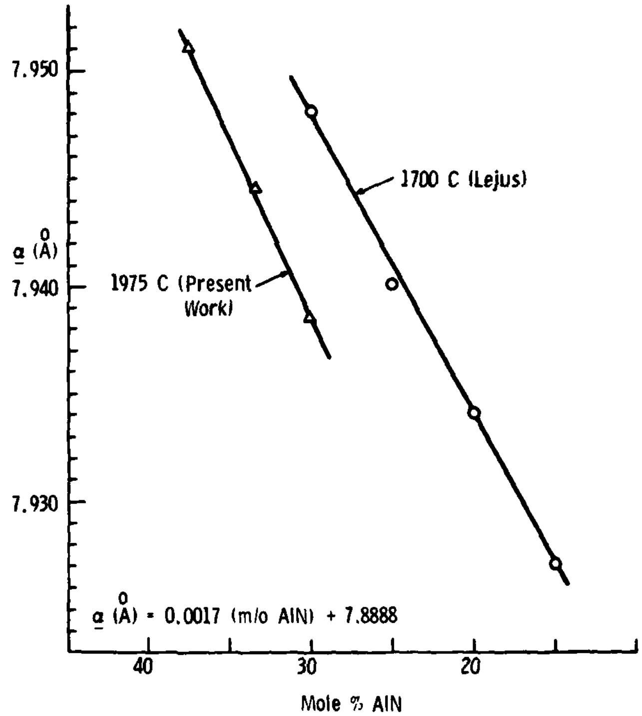

Figure 4. *Refined lattice parameters* of cubic aluminum oxynitride spinel (ALON) as a function of mole % AlN at 1975 C.

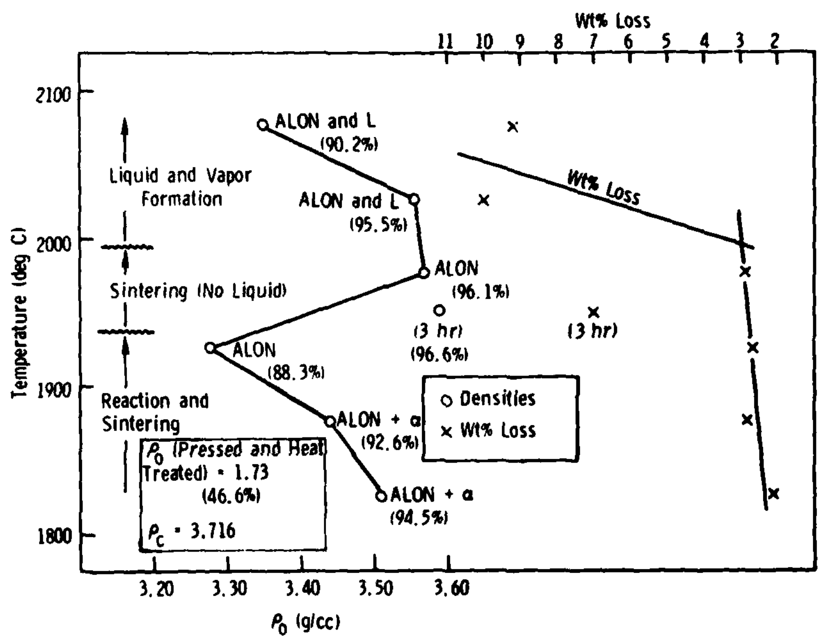

Figure 5. Variation of density and weight percent loss with temperature (1-hr runs) of 37.5 mole % AlN mixtures.

A preliminary apparent decrease in density occurs during formation of ALON, caused by the diminishing amount of the higher density α-Al_2O_3. Optimum properties and liquidless sintering must be carried out below about 2000 C to prevent the formation and subsequent vaporization of a liquid phase. Residual, quenched liquid, either as a noncrystalline phase or as one of the "AlN" polytypes, will have a large effect on the properties of the material. Note the increase in density for the material sintered for three hours.

Formation of sintered, single-phase ALON ceramics free of second phases can be easily accomplished in the single-phase field. However, detailed reflected and transmitted light microscopy of sintered ALON samples do reveal occasional evidence of grain boundary phases - probably low melting phases due to unavoidable impurities in the starting powders, especially AlN. Figure 6 illustrates a typical microstructure of material exhibiting a relatively large amount of remnant porosity. This material seems to react and sinter quite rapidly into a uniform grain size microstructure. For our experimental conditions, grain growth is quite rapid, with an average size somewhere between 50 and 100 μm. An abundance of apparent spinel-law ({111}) twins is also evident in Figure 6b, which seem to polish at different rates, since some are elevated and others are recessed. A small increase in sintering temperature (1975 C to 2025 C) results in much reduced residual porosity and a microstructure that only microscopic defocussing at low magnification (Figure 7a) will resolve the grain boundaries; scanning electron micrographs of fracture surfaces of this same material are illustrated in Figures 7b and c to further illustrate the microstructure.

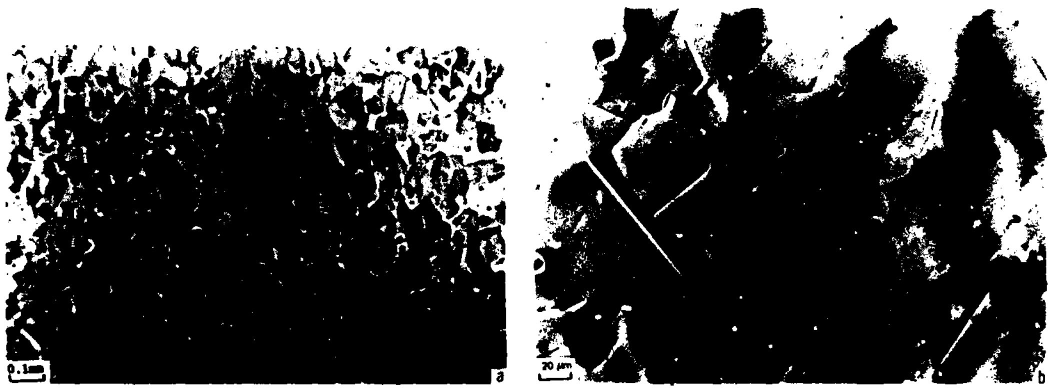

Figure 6. Microstructures of single-phase cubic aluminum oxynitride spinel; T = 1975°C; 30 mole % AlN; t = hour.

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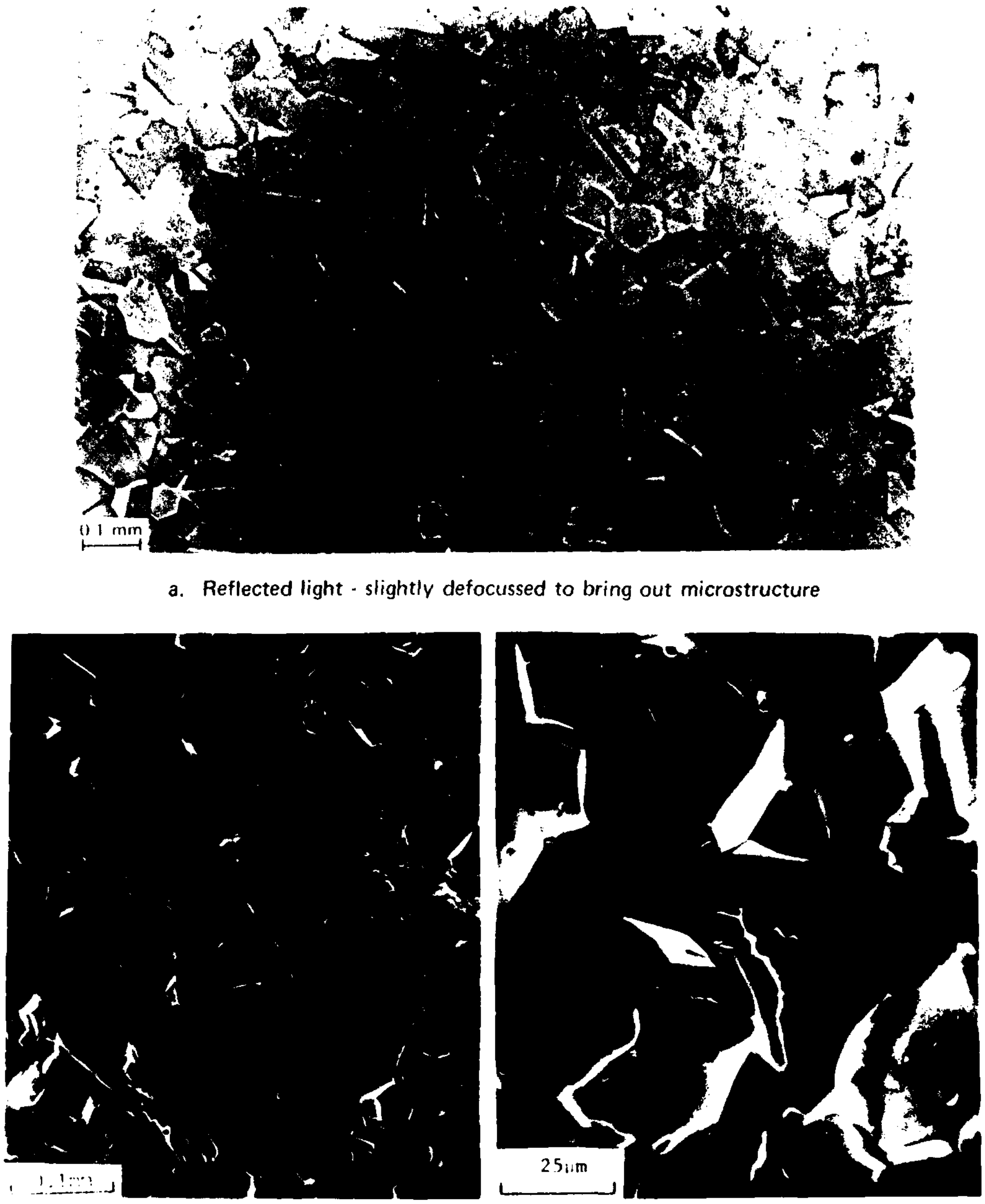
IV. PROPERTY DETERMINATIONS

Preliminary properties are now being measured on sintered ALON materials. Figure 8 illustrates a polished disk (0.035" thick) of a 30 mole % AlN material exhibiting a high degree of transparency; the word ALON is behind the disk. Table 1 lists several of the properties of ALON which have been measured. Of particular interest in terms of a multimode sensor window is the high hardness, relatively high IR cutoff and the excellent dielectric properties. Table 2 is a more extensive listing of the dielectric properties and Figure 9



a. Reflected light - slightly defocussed to bring out microstructure

b. SEM - fracture surface

c. SEM - fracture surface

Figure 7. Microstructures of single-phase cubic aluminum oxynitride spinel; T = 2025 C; 30 mole % AlN; t = 1 hour.

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illustrates the dielectric constant and loss tangent as a function of temperature for measurements at 10 MHz frequency. These properties will significantly improve where 100% of theoretical density is achieved. Figure 10 illustrates the excellent oxidation resistance of ALON in air up to 1200 C. At this temperature a protective film is formed that serves to persist and inhibit additional oxide formation.

Figure 8. Transparent disk of single-phase cubic aluminum oxynitride spinel.

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Table 1. PROPERTY DATA FOR CUBIC ALUMINUM OXYNITRIDE SPINELS

Mole% AlN	Density g/cc	Max% Density	Sonic Modulus $\times 10^6$ psi	Hardness Knoop(100) kg/mm^2	K_{Ic} MN/m$^{3/2}$	κ' (10 MHz) (25 C)	Tan δ (10 MHz) (25 C)	IR Cutoff (μm)	Ref. Index (λ=0.55 μm)
30	3.66	98.8	46.03	1788	0.48	8.56	0.0004	5.18	1.770
33.3	3.68	99.2							
37.5	3.57	96.1		1624					1.785

K_{Ic} = Critical stress intensity factor from Vickers hardness indent
κ' = Dielectric constant
Tan δ = Dielectric loss tangent

Table 2. DIELECTRIC PROPERTIES OF ALON

T°C	Freq., Hz	10^2	10^3	10^4	10^5	10^6	10^7
25	κ'	8.56	8.56	8.56	8.56	8.56	8.56
	κ''	0.013					
	tan δ	0.0015	0.0011	0.0006	0.0005	0.0005	0.0004
	σ	7.1×10^{-13}					
150	κ'	8.62	8.60	8.60	8.60	8.60	8.60
	κ''	0.028					
	tan δ	0.0033	0.0037	0.0024	0.0018	0.0010	0.0006
	σ	1.6×10^{-12}					
300	κ'	8.97	8.79	8.72	8.65	8.64	8.64
	κ''	0.242					
	tan δ	0.0270	0.0108	0.0044	0.0029	0.0026	0.0021
	σ	1.343×10^{-11}					
400	κ'	10.0	9.40	9.13	8.95	8.76	8.72
	κ''	0.709					
	tan δ	0.0709	0.0495	0.0229	0.0078	0.0037	0.0031
	σ	3.94×10^{-11}					
500	κ'	14.0	11.7	9.92	9.18	8.95	8.87
	κ''	14.2	2.48				
	tan δ	1.014	0.212	0.0941	0.0370	0.0136	0.0070
	σ	7.89×10^{-10}	1.4×10^{-9}				

κ' = Dielectric constant
κ'' = Relative loss factor
tan δ = Loss tangent = κ''/κ'
σ = Dielectric conductivity

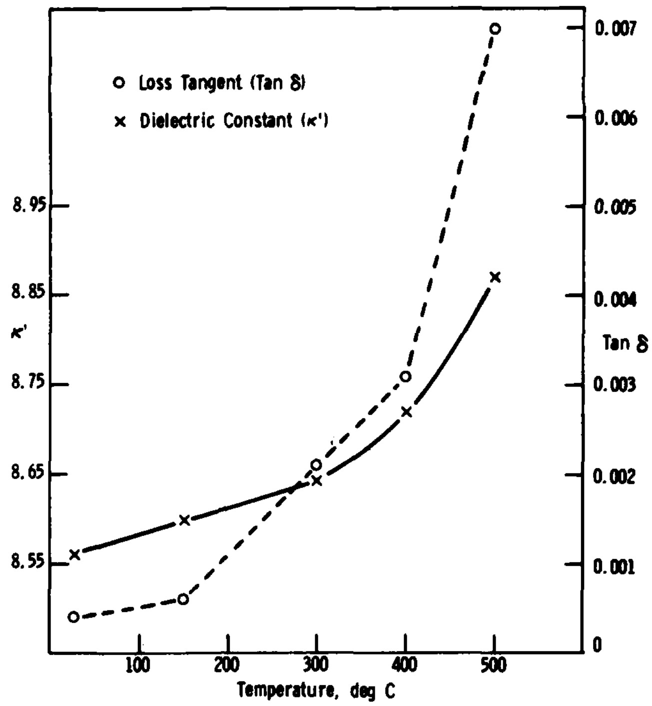

Figure 9. Dielectric properties of 30 mole % AlN ALON measured at 10 MHz.

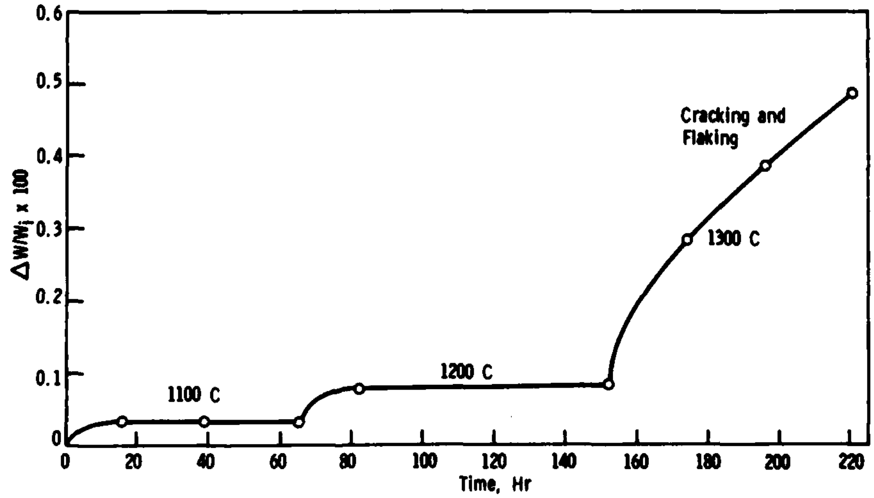

Figure 10. Oxidation of 30 mole % AlN ALON at various temperatures as a function of time in air.

Thermal expansion has also been measured and is illustrated in Figure 11; note that the sample returned to its original exact dimension. The average coefficient of thermal expansion (α) calculated from 20 C to 980 C is 7×10^{-6} $C^{\circ-1}$, significantly lower (22%) than α-Al_2O_3 at 9×10^{-6} $C^{\circ-1}$ at equivalent temperatures. This value suggests a much superior thermal shock resistance of this material over α-Al_2O_3. Many preliminary shapes and sizes of ALON have been

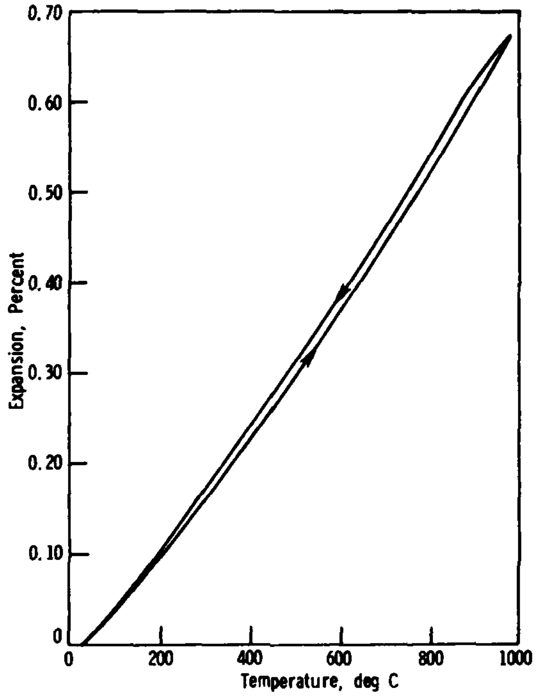

Figure 11. Thermal expansion of 30 mole % AlN ALON.

fabricated. These are illustrated in Figure 12. The thin disk covering the AMMRC logo was simply saw cut and was not polished at all.

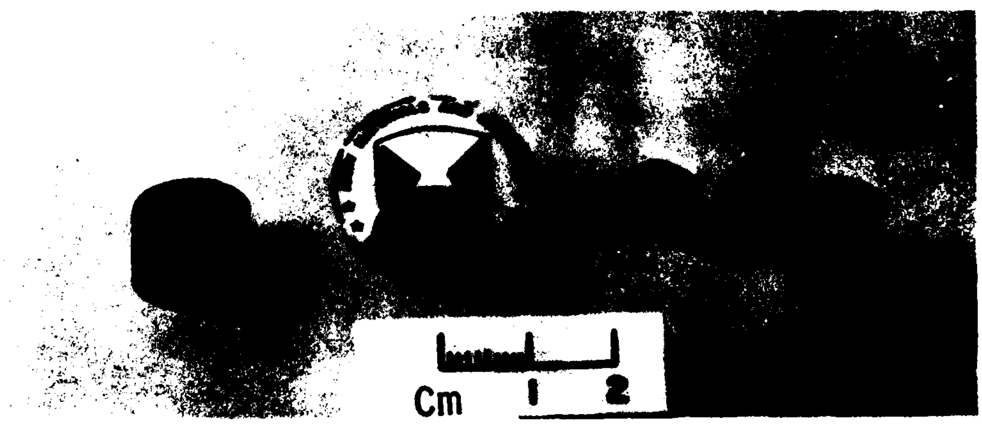

Figure 12. Various fabricated bodies of ALON.
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V. SUMMARY AND CONCLUSIONS

A refined high temperature phase diagram in the region of stability of cubic aluminum oxynitride spinel (ALON) along the Al_2O_3-AlN composition join has been determined. This material can also be

described as nitrogen-stabilized cubic aluminum oxide. Using this newly determined diagram, single-phase ALON has been reactively sintered to nearly full density. Sintering is carried out quite easily and polished thin disks exhibit visible light transparency. The lattice parameter of ALON varies with composition from 7.938 Å for 30 mole % AlN to 7.951 Å for 37.5 mole % AlN sintered at 1975 C. At this temperature the limit of ALON crystalline solution is from 40 to about 27 mole % AlN.

Preliminary properties determined on ALON strongly suggest that it is a viable candidate for future multimode EM sensor window requirements and as an improved substitute for Al_2O_3 in many other applications.

VI. ACKNOWLEDGMENT

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The stringent mechanical/thermal and broadband electromagnetic wave transmission requirements for sensing mechanisms of the future require optimized and new material concepts. Aluminum oxide-based ceramics and single crystals are strong contenders for these applications, but exhibit significant directional variation (anisotropy) in properties. This barrier to utilization has been eliminated by an AMMRC-invented material - sintered polycrystalline nitrogen-stabilized cubic aluminum oxide (ALON). This material has been fabricated into dense, transparent bodies with *isotropic* properties: Knoop (100) hardness of 1800, elastic modulus of 46×10^6 psi, a dielectric constant and loss tangent, respectively, at 10 MHz of 8.56 and 0.0004, trivial oxidation in air up to 1200°C, an IR cutoff at 5.2 μm, and an average thermal expansion coefficient (α) of 7.00×10^{-6} $C^{\circ -1}$ (25°C-1000°C). These properties suggest greatly improved performance in many other Al_2O_3 applications. Successful fabrication of ALON was preceded by the determination of the high temperature phase equilibria and crystal chemistry of aluminum oxynitride spinels in the Al_2O_3-AlN system.

Army Materials and Mechanics Research Center,
Watertown, Massachusetts 02172
TRANSPARENT, POLYCRYSTALLINE CUBIC
ALUMINUM OXIDE -
James W. McCauley and Normand D. Corbin

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